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TECHNICAL REPORT

TR-AE-18

(SECRET)

ANALYSIS OF SOVIET THIOKOL SAMPLE

23 JANUARY 1953

PROJECT NO. 30049

AIR TECHNICAL INTELLIGENCE CENTER
WRIGHT-PATTERSON AIR FORCE BASE
DAYTON, OHIO

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TECHNICAL REPORT NO. TR-AE-16

(SECRET)

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Published By

AIR TECHNICAL INTELLIGENCE CENTER
WRIGHT-PATTERSON AIR FORCE BASE
OHIO

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SUMMARY

Purpose

To analyze and evaluate a sample of Soviet produced thiokol fuel tank sealant.

Factual Data

The sample was received as an inclosure with report number ATIC 61102. The information included in that report was as follows:

1. A specimen of the sealing material used in the fuselage integral fuel tank of the Soviet Type 150 experimental aircraft built by former Junkers personnel was obtained and was forwarded for evaluation.

2. Information from other reports on the Type 150 aircraft indicate that it is a two jet engine swept-wing bomber. A large honeycomb type tank is located above the bomb bay (see Figure 1 appendix I). The interior of the tank is constructed with vertical aluminum tubes (see Figure 2 appendix I). Some difficulty was reported in sealing this tank.

The sample as received was stuck between layers of cloth and separation from the cloth was not deemed advisable. It was suggested that a similar size sample of U. S. Thiokol be stuck between layers of similar cloth and comparison tests for low temperature flex be run on both samples.

Discussion

The sample of Soviet produced rubber designated as "thiokol" was a graygreen material. Its soft and gummy texture indicated that it was uncured. The sample as received was stuck between layers of cloth.

In order to derive as much information as possible from this very small sample, the following text procedure was used:

1. Determine the type of polymer and composition by chemical and infra-red spectrographic analysis.

2. Determine the swell characteristics of the elastomer in 100 octane gasoline.

3. Compare qualitatively the low temperature flexibility of the Soviet elastomer with a typical U. S. thiokol.

These tests showed that the polymer had an ether linkage similar to U. S. thiokol B or thiokol D and that an ester type of plasticizer was used. The sample showed a swell of 5.6 per cent after seven days in 100 octane gasoline. Further, the sample was stiff at -40°F and brittle at -65°F. The samples of U. S. thiokol showed approximately the same percent of swell in 100 octane gasoline for seven days. The U. S. samples were flexible at -40°F and -65°F.

Conclusions

The Soviet thiokol had inferior low temperature flexibility when compared with

U. S. composition used for similar purposes. The Soviet sample was very stiff at -40°F and brittle at -65°F, while the U. S. sealants are flexible at these temperatures. It is expected that below -40°F the Soviet sealant would tend to crack with the possibility of leakage resulting.

Infra-red and chemical analysis indicates the Soviet composition to be similar to a U. S. type thiokol which has the polymer structure

$$-CH_2\ CH_2\ OCH_2\ CH_2\ \underset{SS}{\overset{SS-}{\|}}$$

There is evidence that an ester type plasticizer is present. The U. S. thiokol used for aircraft sealant is LP-2 (98% - CH_2 CH_2 OCH_2 CH_2 S_2- and 2% trichloropropane.)

The Soviet sample was equal to the U. S. compositions in resistance to high octane gasoline.

The Soviet sealant appeared to be in an uncured state. In the U. S. these sealants are applied in an uncured state and then allowed to air cure. Sealants so cured are tougher and less vulnerable to damage.

SECTION I

The low temperature flexibility of the Soviet thioelastomer was compared with that of three U. S. -produced thioelastomeric products. Thiokol LP-2 (Thiokol Corporation) and sealants EC-801 and EC-712 (Minnesota Mining and Manufacturing Company). LP-2 is the raw Thiokol polymer used in making the sealants. The results (see Table I) showed that the Soviet material was very stiff at -40°F and brittle at -65°F. The three U. S. products were flexible at both temperatures.

The Soviet material was found to have good resistance to swell. The results in Table I indicate a value of 5.6 per cent when the material was aged in 100 octane gasoline for a period of seven days at 75°F. This value compares satisfactorily with that reported for U. S. -produced thiokols.

Infra-red analysis made from the acetone extract showed that the Soviet thioelastomer had an ether linkage similar to that of Thiokol D ($-CH_2\ CH_2\ OCH_2\ CH_2\ SS-$) which is made from thiokol B by treatment with sodium hydroxide.* It is possible that the Soviet material is a mixture of these two thiokols. In addition to an ether linkage, there is evidence of carbonyl absorption (possibly an ester). There is no evidence of hydroxyl, nitrile, or carbon-to chlorine linkages.

Thioelastomers are prepared by condensation of alkali polysulfides such as sodium polysulfide and various dihalides typified by (1) ethylene dichloride, (2) dichlorodiethyl ethers, and (3) dichlorodiethyl formal. The infra-red data indicate that the Soviet product was probably prepared by condensation of sodium polysulfide or sodium sulfide and dichlorodiethyl ether.

The evidence of carbonyl, possibly an ester grouping, indicates that a plasticizer was probably used in the thio-rubber; however, the size of the sample did not permit positive identification of this material.

Chemical Analysis. Microtechniques were used to determine the total sulfur, carbon, hydrogen, nitrogen, and chlorine percentages in the Soviet thiokol (see Table I). The sulfur determination was also checked using macroanalysis. The oxygen content was obtained by calculation. Since the chemical analysis was determined on the sample as received, it was necessary to correct the values for the filler content. Table II gives the percentages of the various elements present, based on the total sample, i.e., elastomer plus filler; also, Table II shows the values corrected to the basis of the elastomer without filler. Table III shows some U. S. thiokol fubbers and the calculated percentages of the constitutent elements. From Tables II and III, it can be seen, on the basis of chemical analysis, that the Soviet sample corresponds closest to the $-C_2H_4OC_2H_4OC_2H_4\underset{SS}{\overset{}{SS}}-$ polymer unit.

* "Synthetic Rubber", by Barnes, R. B., Leddel, U., and Williams, V. Z., Industrial and Engineering Chemistry (Analytical Edition), Vol 15, pp 83-90, 1943.

TABLE I. SUMMARY OF CHEMICAL ANALYSIS AND PHYSICAL PROPERTIES OF SOVIET-PRODUCED THIOELASTOMER

COMPOSITION[1]	
Rubber content, per cent	59.8
Sulfur, per cent	34.2
Carbon, per cent	19.9
Oxygen, per cent[2]	15.7
Hydrogen, per cent	3.7
Nitrogen, per cent	0.2
Chlorine, per cent	0.1
Ash content, per cent	31.2
Acetone extract, per cent	7.0
PHYSICAL PROPERTIES	
Low-temperature flexibility at:	
-40°F	Very stiff
-65°F	Brittle
Swell in 100 octane gasoline after aging 7 days at 75°F per cent	5.6

(1) Percentages reported were calculated on the basis of the total sample, as received.

(2) Per cent oxygen was calculated by subtracting the sum of the percentages of constitutent elements from 100 per cent.

TABLE II. COMPOSITION DATA FOR SOVIET THIOKOL

Constituent	Amount present(1) in sample as received, per cent	Amount present(2) in elastomer only, per cent
Carbon	19.9	28.9
Hydrogen	3.7	5.4
Sulfur	34.2	49.7
Oxygen	—	15.7(3)
Nitrogen	0.2	0.3

(1) Elastomer plus filler.

(2) Values calculated by dividing per cent of constitutent present (on basis of total sample as received) by the per cent of ash-free material.

(3) Per cent oxygen reported represents the difference found by subtracting the sum of the percentages of constitutent elements from 100 per cent.

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TABLE III. EXAMPLES OF UNIT POLYMERS OF SULFIDES COMPARED WITH SOVIET THIOKOL

Structure(1)	Carbon(2) Per Cent	Hydrogen Per Cent	Oxygen Per Cent	Sulfur Per Cent	REMARKS
$-CH_2CH_2OCH_2CH_2\underset{SS}{SS}-$	24.0	4.0	8.0	64.0	Thiokol B
$-CH_2CH_2OCH_2CH_2SS-$	35.3	5.9	11.7	47.1	Thiokol D
$-CH_2H_4OC_2H_4OC_2H_4\underset{SS}{SS}-$	29.5	4.9	13.1	52.5	—
Soviet Thiokol(3)	28.9	5.4	15.7	49.7	—

(1) The structures shown were taken from a table of unit polymers of sulfides, Martin, S. M, Fr. and Patrick, J. C., "Constitution of Polysulfide Rubbers", <u>Industrial and Engineering Chemistry</u>, Vol. 28, p 1144, 1936.

(2) The percentages indicated were caluculated from the structural formula of a unit of polymer.

(3) The percentage of each constitutent was calculated from the composition data shown in Table I.

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TABLE IV. SPECTROGRAPHIC ANALYSIS OF ASH SAMPLE
FROM SOVIET-PRODUCED THIOKOL

Ingredients[1]	Amount Present in Ash,[2] Per Cent	
Silicon	40	-60
Magnesium	20	-40
Iron	5	-25
Aluminum	0.5	- 5.0
Calcium	0.3	- 3.0
Zinc	0.2	- 2.0
Nickel	0.05	- 0.5
Manganese	0.05	- 0.5
Chromium	0.05	- 0.5
Lead	0.02	- 0.2
Copper	0.02	- 0.2
Barium	0.005	- 0.05
Strontium	0.005	- 0.05
Titanium	0.002	- 0.02
Molybdenum	0.001	- 0.01
Tin	0.001	- 0.01
Antimony	0.001	- 0.01

(1) Ingredients were determined as oxides by spectrographic analysis, but are not necessarily added to or present, as such, in the rubber.

(2) The figures reported throughout the table are ingredient percentage ranges on the basis of the ash.

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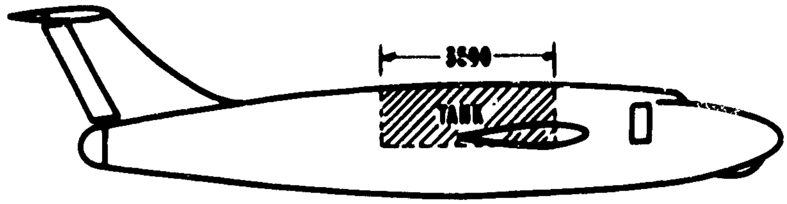
However, infra-red analysis shows that there were some ester groups present. The presence of these groups would reduce the sulfur value and increase the oxygen; the other groupings would change correspondingly. The presence of ester groups would tend to bring the structure closer to that indicated by infra-red analysis, i.e., $-CH_2CH_2OCH_2CH_2SS-$.

SS

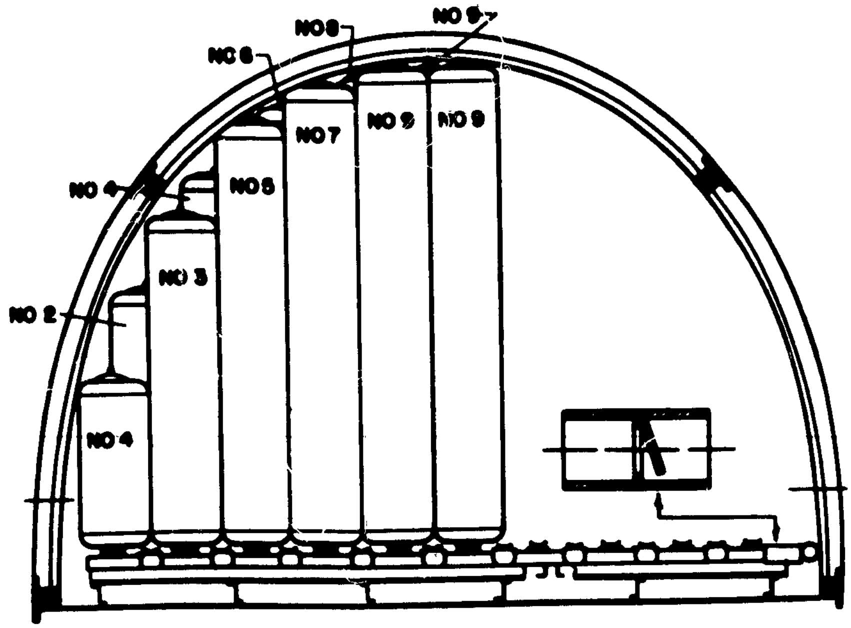
<u>Analysis of Filler Material.</u> A sample of the Soviet sealant material was ashed and found to contain 31.2 percent ash. Table IV summarizes the spectrographic analysis obtained on a very small ashed sample of the Soviet thioelastomer. The principal fillers, reported as oxides, were found to be silicon and magnesium, plus iron which may have been present as an impurity in the filler. These results indicate that magnesium silicate or a clay material probably was used as the primary filler.

APPENDIX I

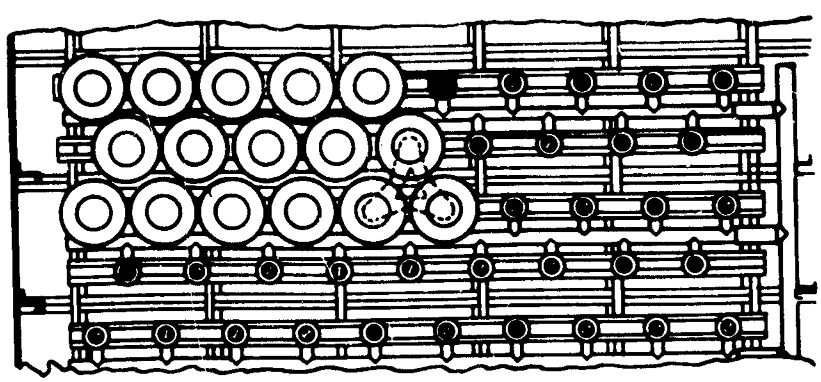


TOTAL LENGTH OF TANK 3500 MM
TOTAL CAPACITY OF TANK [illegible]L

Fig. 1 Sideview of Aircraft



Front View



Top View
Fig. 2 Drawing of Fuel Tank

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DEPARTMENT OF THE AIR FORCE
WASHINGTON, DC

23 June 2010

HAF/IMIO (MDR)
1000 Air Force Pentagon
Washington, DC 20330-1000

Bobby Sammons.
P.O. Box 1680
Cloudcroft, NM 88317-1680

Dear Mr. Sammons

Reference to your letter, undated (attachment 1) requesting a Mandatory Declassification Review (MDR) for Defense Technical Information Center (DTIC) documents:

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AD005736	AD005735
AD006796	AD004876
AD005809	AD003234
AD005808	AD004232

The review for the documents have been completed and the declassification has been downgraded to UNCLASSIFIED and copies are attached for your information.

Address any questions concerning this review to the undersigned at (703) 692-9979 and refer to our case number 07-MDR-076.

Sincerely

JOANNE MCLEAN
Mandatory Declassification Review Specialist

2 Attachments
1. Letter, Request for Documents
2. 10 DTIC Documents

cc: DTIC w/o documents